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Effect of surface preparation on the shear bond strength of orthodontic brackets bonded to zirconia : an in-vitro study

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THE EFFECT OF SURFACE PREPARATION ON THE SHEAR BOND STRENGTH OF ORTHODONTIC BRACKETS BONDED TO ZIRCONIA: AN *IN-VITRO* STUDY

NATHANIEL WIEDER, D.M.D.

A Thesis Presented to the Faculty of the College of Dental Medicine of Nova Southeastern University in Partial Fulfillment of the Requirements for the Degree of

MASTER OF SCIENCE

December 2015

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I certify that I am the sole author of this thesis, and that any assistance I received in its preparation has been fully acknowledged and disclosed in the thesis. I have cited any sources from which I used ideas, data, or words, and labeled as quotations any directly quoted phrases or passages, as well as providing proper documentation and citations. This thesis was prepared by me, specifically for the M.S. degree and for this assignment.

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Dedication

To my family for their unwavering support throughout my educational journey

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Abstract

THE EFFECT OF SURFACE PREPARATION ON THE SHEAR BOND STRENGTH OF ORTHODONTIC BRACKETS BONDED TO ZIRCONIA: AN *IN-VITRO* STUDY

DEGREE DATE: DECEMBER 18, 2015

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Objectives: The purpose of this *in-vitro* study was to evaluate the effects of three different surface preparation methods on the shear bond strength of orthodontic brackets bonded to zirconia and determine the most appropriate method. **Methods:** 45 zirconia and 30 leucite-reinforced porcelain mandibular premolar crowns were divided into 5 groups and received the following surface preparations: 37% phosphoric acid and non-hydrolyzed silane, 4% hydrofluoric acid and hydrolyzed silane, microetch with 50μ Al_2O_3 particles. A universal adhesive primer containing MDP was applied and the brackets were bonded with a bis-GMA composite resin. Shear bond strength (SBS) at bond failure and ARI score were recorded. **Results:** There was a statistically significant difference among the studied groups for the SBS. The highest mean SBS (11.03 MPA) was recorded for the zirconia/microetch group, and the lowest SBS (3.49 MPa) for the

zirconia/phosphoric acid group. The leucite-reinforced porcelain/ hydrofluoric acid group had significantly more fractures than any other debond pattern. The zirconia/hydrofluoric acid group was the only one with a SBS (8.08 MPa) that fell within the recommended range of 6-8 MPa. This group also had a favorable debond pattern with most composite remaining on the bracket.

Conclusions: Important consideration should be given to the surface preparation of porcelain and zirconia prior to bonding orthodontic attachments. Phosphoric acid etch is not an adequate surface preparation when bonding to zirconia. Hydrofluoric acid is not suitable when bonding to leucite-reinforced porcelain, as it is associated with a higher rate of surface fracture. Microetch with 50μ Al_2O_3 particles in combination with an MDP containing universal adhesive primer provided optimal mean shear bond strength, along with favorable debond patterns when bonding to zirconia. Hydrofluoric acid etch in combination with a silane and a universal primer containing MDP provided acceptable shear bond strength to zirconia. This protocol was not significantly different from zirconia prepared with microetch and either method can be successfully employed.

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Chapter 1: Introduction

1.1 Adhesive Bonding in Orthodontics

Adhesive bonding plays an integral role in today's practice of orthodontics. Before the advent of adhesive bonding, banding teeth with welded brackets was necessary in order to gain adequate control for orthodontic movements[1]. The advantages of directly bonded attachments include superior esthetics, improved oral hygiene, and more efficient chair time[2, 3].

Buonocore[4] pioneered dental adhesive bonding when he proposed that acid etching dental surfaces increases surface energy and area, promoting increased bond strength between acrylic and tooth. The demineralizing action of the acidic etchant enables the distribution of polymerizable monomers into and around hydroxyapatite prisms[5]. Gwinnet and Matsui[6] described the finger-like projections of adhesive into microporosities, or "resin tags", and suggested that these adhesive extensions are the primary mechanism of micromechanical bond between tooth and adhesive. Newman[7] was the first to bond orthodontic attachments directly to enamel utilizing concentrated phosphoric acid and epoxy as an adhesive.

1.2 Adhesive Bonding to Porcelain

The number of adults seeking comprehensive orthodontic treatment is increasing, resulting in situations where bonding to ceramic restorations may be necessary[8]. Studies by Paffenbarger *et al*.[9] and Moffa *et al*.[10] were of the first that successfully demonstrated adhesive bonding to porcelain in dentistry using only a chemical coupler to bond porcelain teeth to denture bases. A study by Ghassemi-Tary[11] fostered porcelain bonding in orthodontics when he demonstrated that acceptable bond strengths can be achieved

between orthodontic attachments and porcelain. Subsequently, Newman[12] described the preparation of porcelain surfaces with pumice prior to application of the chemical coupler and bonding. Calamia and Simonsen[13] showed that preparation of porcelain with acidic etch before coupler application increased shear bond strength between the surface and resin composite.

1.3 Surface Preparations

There are several methods for enhancing ceramic surface receptiveness, including mechanical preparation, chemical preparation, or a combination of both[8, 14].

1.3.1 Phosphoric Acid

Phosphoric acid (H_3PO_4) is commonly used as a surface preparation agent in dentistry[15]. Phosphoric acid chemically removes surface glaze, and neutralizes the alkalinity of the porcelain in preparation for silane primer application[16]. The application of phosphoric acid does not produce microporosities that yield mechanical retention to porcelain[17]. There is uncertainty regarding the use of phosphoric acid as an adequate etchant for porcelain. In a study that evaluated the effects of a self-etching primer on feldspathic porcelain, Ajlouni *et al*.[14] found that phosphoric acid alone produced significantly lower shear bond strengths than other surface preparation techniques. Studies by Lifshitz *et al*.[18] and Bourke *et al*.[16] reported that phosphoric acid in combination with silane primer provided adequate shear bond strength when bonding to porcelain.

1.3.2 Hydrofluoric Acid

Hydrofluoric acid (HF) removes surface glaze and chemically creates microporosities in the porcelain surface by removing the glass phase in the porcelain

matrix[16]. Etching time and etchant concentration are factors that affect the effectiveness of hydrofluoric acid[19]. Gillis *et al.*[20] reported that hydrofluoric acid produced significantly higher shear bond strengths than other methods when bonding to porcelain. Another study by Bourke *et al*.[16] found hydrofluoric acid caused the most surface damage at bracket debonding[14]. Larmour *et al*.[21] showed no significant difference in shear bond strength between hydrofluoric and phosphoric acid, questioning the use of hydrofluoric acid intraorally due to its toxicity and potentially harming affects on periodontal tissue. In a study that evaluated the effects of surface preparation on different ceramics, Karan *et al*.[22] showed that hydrofluoric acid combined with a silane coupler produced significantly lower bond strength when bonding to leucite-reinforced porcelain.

1.3.3 Microetch

Microetch removes surface glaze and mechanically creates microporostites in the porcelain surface[20]. Sandblasting pressure, particle size, and particle shape are factors that affect the effectiveness of microetching[19]. Gillis *et al.*[20] showed that microetch produced adequate bond strength, however produced significant surface damage at bracket debonding. Schmage *et al.*[23] found that microetch alone produced significantly lower bond strengths and higher surface damage than other methods. Another study found microetch, followed by hydrofluoric acid and silane coupler to provide optimal shear bond strength[14].

1.3.4 Silane

Silanes increase adhesion to porcelain by chemically coupling porcelain with adhesive. The hydrolyzable group of the silane reacts with the silica in the porcelain

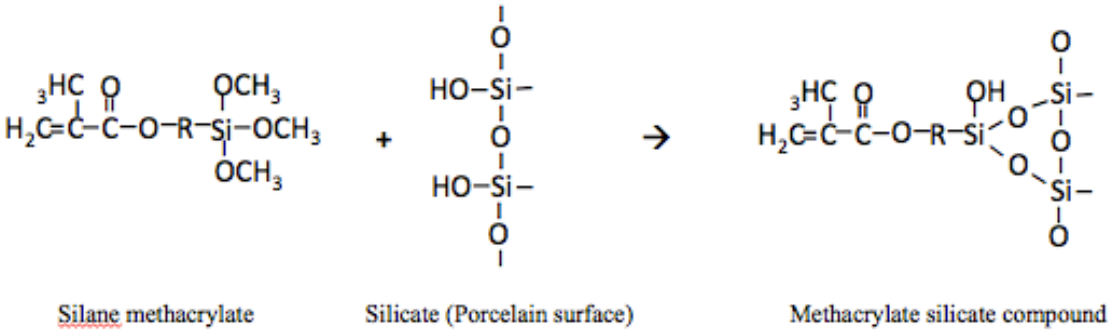


Figure 1. Silane-porcelain chemical reaction

substrate, while the organic group of the silane is free to react with the adhesive[12] (Figure 1). Newman *et al.*[24] found that silane enhances the bond strength when bonding to feldspathic porcelain[24]. Bourke *et al.*[16] showed that silane application was the most important factor in achieving adequate bond strength compared to other preparation methods.

1.3.5 Phosphate Ester

Studies have shown that traditional bonding techniques are not as effective when bonding to zirconia compared to ceramics. Kern and Wegner[25] showed that the use of silane did not significantly increase bond strength and was ineffective due to the absence of silica in zirconia. Kern and Wegner[25] and Wolfart *et al.*[26] found that the use of a primer containing phosphate monomer significantly increased the bond strength to zirconia and resulted in successful bond stability. Blatz *et al.*[27] determined the shear bond strength with a phosphate monomer primer to be superior to preparation with conventional acid etch and silane. The agent, 10-methacryloyloxydecyl dihydrogen phosphate (MDP), a phosphate ester, has been shown to chemically bond to zirconia. The phosphate ester group of the adhesive monomer bonds directly to metal oxides, resulting

in a chemical bond between MDP and zirconium oxides[25, 26] (Figure 2). The organic group of the phosphate ester is then free to react with the adhesive.

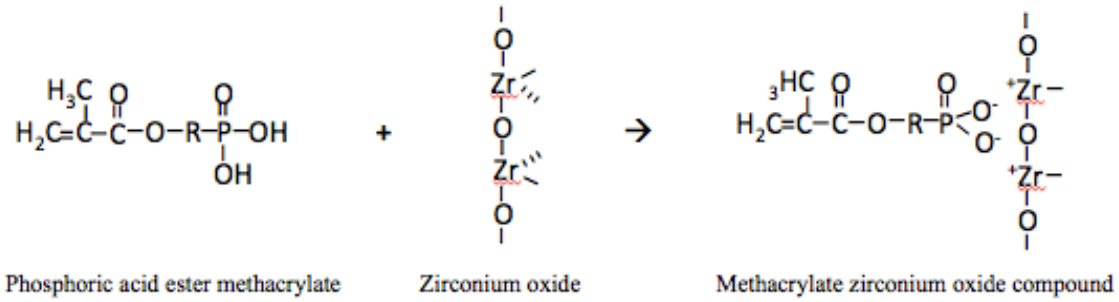


Figure 2. Phosphate ester-zirconia chemical reaction

1.3 Leucite Reinforced Porcelain

Leucite ($KAlSi_2O_6$) is incorporated into feldspathic porcelain in order to reinforce the primarily silica-based structure. When heated to 820°C, leucite undergoes a phase transformation from tetragonal to cubic form, which has a lower coefficient of thermal expansion[28]. Leucite reinforced porcelain is a commonly used restorative material due to its resemblance and similar wear properties to natural tooth structure[29].

1.4 Zirconia

Zirconia (ZrO_2) is the crystalline dioxide form of zirconium (Zr) and is classified as a metal oxide. The crystalline structure of zirconia can be arranged in tetragonal, cubic, and monoclinic forms[30]. Yttria (Y_2O_3) is an oxide that is added to zirconia to partially stabilize the tetragonal form. When stress is applied to the zirconia surface, phase transformation from tetragonal form to monoclinic form occurs inducing micro-cracks that lead to wear over time[31]. The yttria stabilized tetragonal zirconia polycrystalline (Y-TZP) compound has gained popularity as a restorative material

because of its biocompatibility, strength, wear resistance, steel-like modulus of elasticity, and resistance to corrosion[31]. A 2014 survey by the National Association of Dental Laboratories reported that 18% of all crown and bridge restorations were fabricated in zirconia[32].

1.5 Shear Bond Strength

The most suitable surface conditioning technique is one that results in satisfactory shear bond strength between substrate and orthodontic bracket[14]. The *optimal* shear bond strength should be strong enough to withstand intraoral forces during orthodontic treatment, yet weak enough to induce only minimal surface damage during the debonding procedure[16]. Studies have shown several variables such as crosshead speed[33], the use of precoated brackets[34], and thermocycling[35] to have a significant effect on the shear bond strength. Consequently, one study suggested that shear bond strength is best implemented as a comparative ranking scale to compare different surface preparations[36]. Another study recommended that *in-vitro* shear bond strength should not be directly extrapolated to *in-vivo* conditions[37].

1.6 Adhesive Remnant Index

The Adhesive Remnant Index (ARI) is used to classify the site and type of bond failure between substrate, adhesive, and bracket[38]. The ARI is measured on a scale of 0-3: 0-No adhesive left on surface, 1-Less than 50% of the adhesive left on the surface, 2-More than 50% of the adhesive left on the surface, 3-All adhesive left on surface with distinct impression of bracket mesh. The ARI has been reported to have a weak to no correlation with shear bond strength[16, 37, 39].

1.7 Purpose

The purpose of this *in vitro* study is to evaluate the effects of three different surface preparation methods on the shear bond strength of orthodontic brackets bonded to zirconia and determine the most appropriate method. There is limited literature discussing the ideal surface preparations when bonding orthodontic brackets to zirconia. Whitlock *et al.*[40] demonstrated adequate shear bond strength to feldspathic porcelain to be between 6-8 MPa. This study is significant in that it will determine if surface preparation has a significant effect on shear bond strength when bonding to zirconia. This study will also determine if surface preparation has a significant damaging effect on zirconia when debonding orthodontic brackets. The results of this study will provide the orthodontist with an optimal protocol for bonding orthodontic attachments to zirconia.

1.8 Specific Aims

1.8.1 To measure the shear bond strength of orthodontic brackets bonded to leucite-reinforced porcelain and zirconia when using different surface conditioning techniques.

1.8.2 To measure the surface damage caused when debonding orthodontic brackets bonded to leucite-reinforced porcelain and zirconia.

1.9 Location of Study

The design, preparation, data collection and data analysis of this study took place at:

Nova Southeastern University College of Dental Medicine
3rd Floor Biomaterials Laboratory
3200 South University Drive
Fort Lauderdale, Florida 33328

Chapter 2: Materials and Methods

2.1 Study Description

Based on a power analysis, 45 zirconia crowns and 30 leucite-reinforced porcelain crowns were used for this *in-vitro* study.

2.1.1 IRB Approval

IRB approval to conduct this research was not required. There was no protected information or human/animal subjects or tissues used for this study.

2.1.2 Ethical Issues

No potential ethical issues were identified as part of this research study.

2.1.3 Grant

This study was awarded a grant by the Health Professions Division at Nova Southeastern University.

2.2 Sample Size

The sample size was determined based on similar studies by Ajlouni *et al*.[14] and Lifshitz *et al*.[18] that showed statistical significance using 15 samples per group. For a study power of 80%, using an alpha of 5% and a standardized effect size of 0.5, 15 samples per group was considered appropriate.

2.3 Sample Preparation

A prefabricated plastic lower right second premolar (#29) prepared for a full coverage porcelain crown was secured into a typodont (Figure 3). The lower right segment and opposing teeth were sprayed with titanium dioxide than scanned with a Cerec Bluecam scanner (Figure 4) (Sirona Dental, Inc., *Charlotte, North Carolina*). The

CEREC® Connect software Version 4.2.3 (Sirona Dental, Inc., *Charlotte, North Carolina*) was used to scan and design the full coverage crown (Figure 5). The scan and crown design were sent to DSG Clearwater laboratory (*Clearwater, FL*) for fabrication of 45 identical monoclinic zirconia crowns and to Comprehensive Dental Studio Inc. (*Davie, FL*) for fabrication of 30 identical leucite reinforced porcelain crowns.

Figure 3. Prefabricated crown preparation

Figure 4. Scanning typodont

Ten leucite reinforced porcelain crowns and ten zirconia crowns were randomly selected for inspection under 20x magnification to confirm that the samples were identical. The crowns were assessed by the principle investigator for uniform surfaces and evenly distributed surface glaze (Figures 6,7).

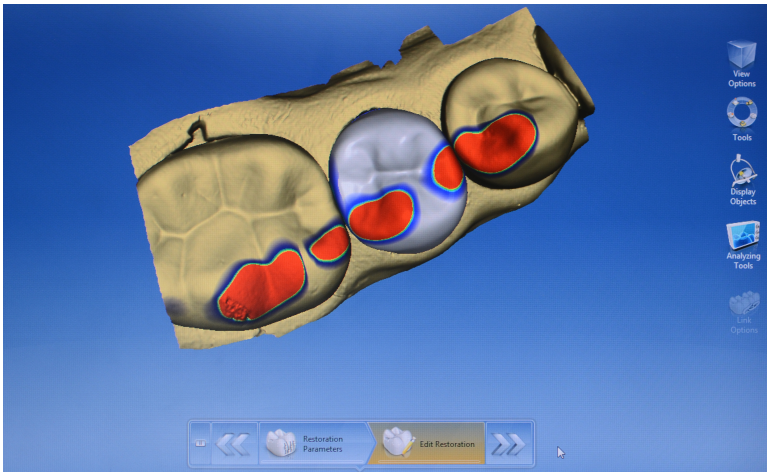


Figure 5. Computer aided crown design

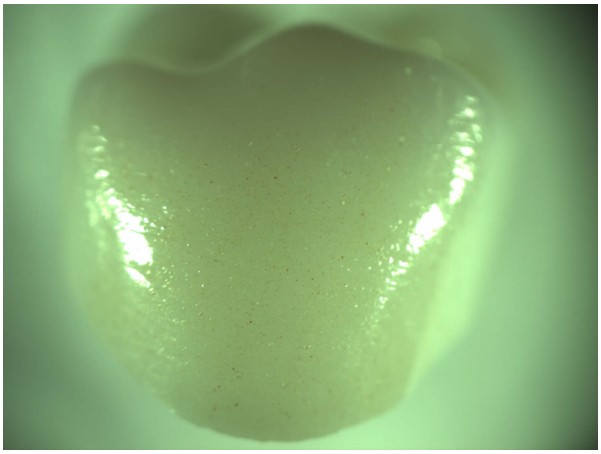


Figure 6. Buccal surface of zirconia crown (20x)

All samples were mounted with Type IV dental stone using silicone templates. The facial surface of the crowns was left exposed 3mm above the stone for access to bond orthodontic brackets (Figure 8).

The samples were randomly assigned to 5 groups and received the following surface preparation protocols:

Group I: 15 leucite-reinforced porcelain samples were etched for 4 minutes with 37% phosphoric acid (Reliance Orthodontic Products, Inc., *Itasca, IL*). Non-hydrolized silane primer (ORMCO™, *Orange, CA*) was applied for 60 seconds without removal of the

acidic etchant. The surface was rinsed for 5 seconds and air-dried with compressed oil-free air (Figure 9).

Group II: 15 zirconia samples were etched for 4 minutes with 37% phosphoric acid (Reliance Orthodontic Products, Inc., *Itasca, IL*). Non-hydrolized silane primer (ORMCO™, *Orange, CA*) was applied for 60 seconds without removal of the acidic etchant. The surface was rinsed for 5 seconds and air-dried with compressed oil-free air (Figure 9).

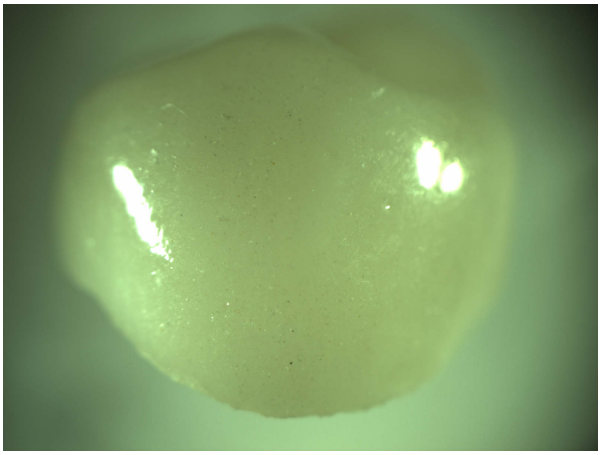


Figure 7. Buccal surface leucite reinforced porcelain crown (20x)

Figure 8. Mounted crown

Group III: 15 leucite-reinforced porcelain samples were etched for 4 minutes with 4% hydrofluoric acid (Reliance Orthodontic Products, Inc., *Itasca, IL*). The surfaces were rinsed for 5 seconds and air-dried with compressed oil-free air, then hydrolized silane primer (Reliance Orthodontic Products, Inc., *Itasca, IL*) was applied for 60 seconds (Figure 10).

Group IV: 15 zirconia samples were etched for 4 minutes with 37% phosphoric acid (Reliance Orthodontic Products, Inc., *Itasca, IL*). Non-hydrolized silane primer (ORMCO™, *Orange, CA*) was applied for 60 seconds without removal of the acidic etchant. The surface was rinsed for 5 seconds and air-dried with compressed oil-free air

Group V: 15 zirconia samples were microetched with 50μ Al_2O_3 particles for 5 seconds at 100 psi (Figure 11). The surface was rinsed for 5 seconds and air-dried with compressed oil-free air

Assure®Plus (Reliance Orthodontic Products, Inc., *Itasca, IL*), a universal adhesive primer that contains 10-methacryloyloxydecyl dihydrogen phosphate (MDP) was applied to the samples. As per the manufacturer's directions, the primer was light cured for the zirconia groups and air-dried for the leucite-reinforced porcelain subgroups. APC™ II adhesive coated brackets (3M Unitek, *Monrovia, CA*) were used in order to standardize the amount of composite used for all samples. The brackets were pressed to the sample surfaces with 300 grams of force using a Dontrix gauge (Orthopli, *Philadelphi, PA*) to standardize the amount of force used for all samples (Figure 12). Excess composite was removed with a fine explorer. The bis-GMA composite was light cured using a VALO® Ortho curing light (Ultradent Products, Inc., *South Jordan, UT*) for 3 seconds on mesial and distal sides. A Dementron® L.E.D. radiometer (Kerr Corporation, Orange, CA) was

used to ensure the light was curing at a constant output of 1000mW/cm^2. Samples were then thermocycled between 5°C and 55°C for 500 cycles (65 seconds per cycle, 30 seconds dwell time, 5 second transfer time), then stored in 37°C water as per International Organization for Standardization standards[41].

Figure 9. Bonding protocol for Groups I and II

Figure 10. Bonding protocol for Groups III and IV

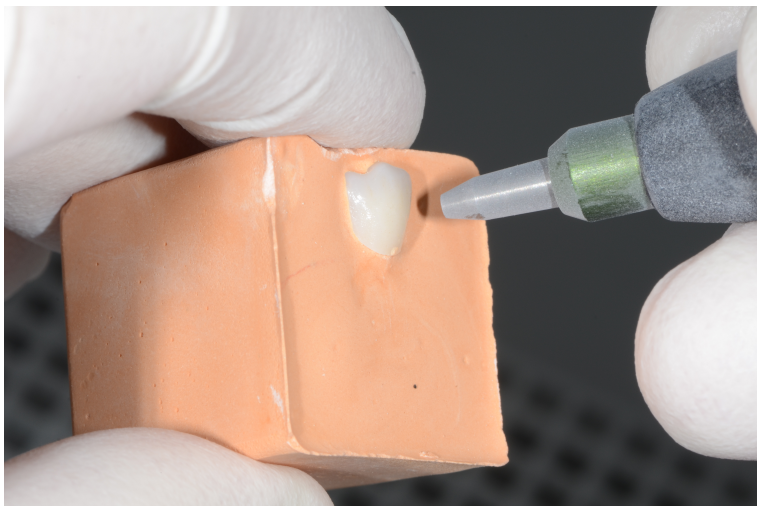


Figure 11. Bonding protocol for Group V

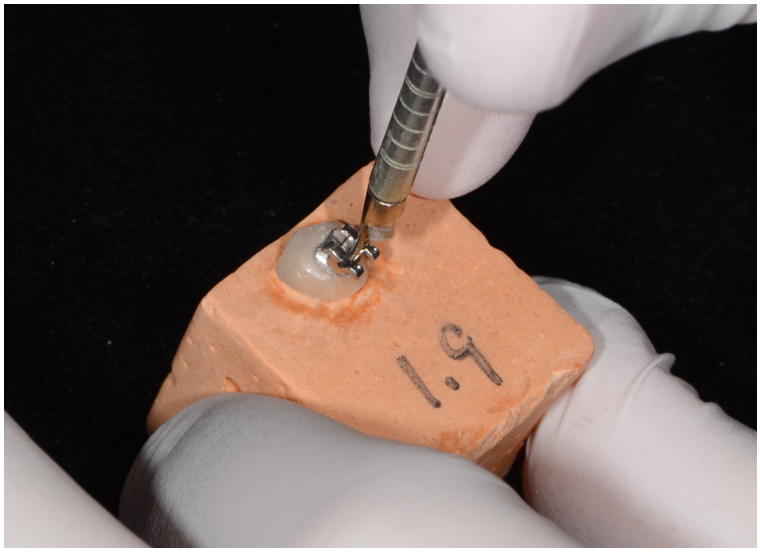


Figure 12. Standardized pressure application

2.4 Experiment

Brackets were debonded using a Universal Testing Machine (*Instron*, Grove City, PA). The blade was perpendicularly oriented to the bracket base and an occluso-gingival force was applied at a crosshead speed of 5mm/min [14, 16] (Figure 13). The shear bond strength at the time of bond failure was recorded in Newtons and converted into MPa (N/cm^2). Samples were examined under a microscope at 20x magnification, and the Adhesive Remnant Index was recorded based on the following modified scale as used by Larmour

et al[21].: 0-No adhesive left on surface, 1-Less than half of the adhesive left on the surface, 2-More than half of the adhesive left on the surface, 3-All adhesive left on surface with distinct impression of bracket mesh, 4-Porcelain surface fracture.

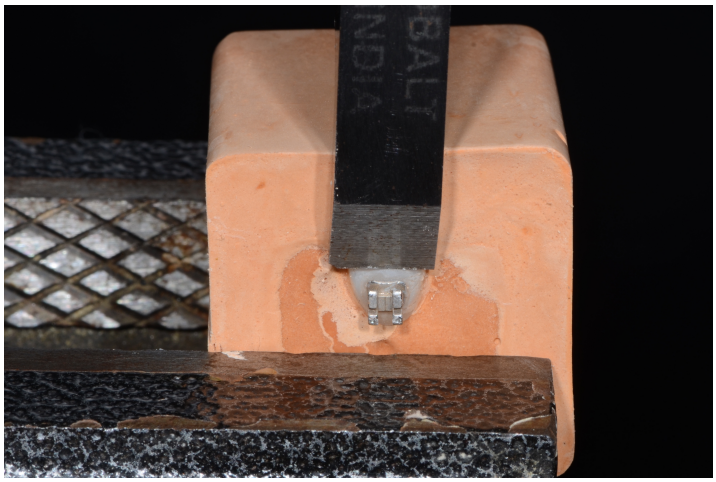


Figure 13. Sample mounted in universal testing machine

2.5 Data Storage

Data was recorded and stored using Microsoft® Excel® for Mac 2011, Version 14.4.8 (Microsoft, *Redmond, WA*).

2.6 Statistical Analysis

The mean, median, and distribution of shear bond strength at bond failure were determined for each group (Table 1). A 1-way analysis of variance (ANOVA) was used to compare means across groups and a pairwise comparison using Tukey's HSD test was implemented. A Chi-square test using Monte-Carlo simulation was employed to examine differences by ARI scores. Significant differences were determined by examining each cell standardized residual.

Chapter 3: Results

The descriptive statistics for the shear bond strengths of the five groups are provided in Table 1. Three samples were lost due to premature debond caused by operator error. There was a significant effect of Group on MPa at the $p<0.05$ level [$F(2, 67) = 13.09$, $p = 0.001$]. Pairwise comparisons of groups are provided in Table 2. Figure 14 demonstrates the mean shear bond strength per group. Groups *not* connected by the same letter are significantly different.

- Group I - AB
- Group II - A
- Group III - C
- Group IV - BC
- Group V - C

Descriptive statistics for the percentage of ARI score per group are provided in Table 1. Results from the chi-square test reveal the percentage of ARI scores differed by group, $c^2(16, N = 72) = 108.70$, $p = 0.001$ (Figure 15). Visual assessments of ARI scores are provided in Figure 16.

Specific differences are as follows:

- Group I - had significantly more ARI scores of 0
- Group II - had significantly more ARI scores of 0
- Group III - had significantly more ARI scores of 4
- Group IV - had significantly more ARI scores of 1
- Group V – had significantly more ARI scores of 1, 2, 3,

Table 1.

Descriptive Statistics

		N	Mean	SD	Min	Max
Group	Group 1	15	4.89	2.96	1.35	11.72
	Group 2	14	3.49	2.34	0.72	8.13
	Group 3	14	9.80	3.26	5.05	18.12
	Group 4	14	8.08	4.72	1.09	16.14
	Group 5	15	11.03	3.12	5.65	15.64

		ARI - 0	ARI - 1	ARI - 2	ARI - 3	ARI - 4
Group	Group 1	13 (87%)	1 (7%)	0 (0%)	0 (0%)	1 (7%)
	Group 2	13 (93%)	1 (7%)	0 (0%)	0 (0%)	0 (0%)
	Group 3	0 (0%)	1 (7%)	0 (0%)	0 (0%)	13 (93%)
	Group 4	5 (36%)	8 (57%)	1 (7%)	0 (0%)	0 (0%)
	Group 5	1 (7%)	8 (53%)	2 (13%)	4 (27%)	0 (0%)

Table 2.

Pairwise Comparisons

Group		Group	Difference	Lower 95% CI	Upper 95% CI	P-Value
Group II	vs.	Group I	-1.40	-4.90	2.11	0.80
Group III	vs.	Group I	4.91	1.40	8.41	*0.00
Group IV	vs.	Group I	3.19	-0.32	6.69	0.09
Group V	vs.	Group I	6.14	2.69	9.58	*0.00
Group III	vs.	Group II	6.31	2.74	9.87	*0.00
Group IV	vs.	Group II	4.59	1.02	8.15	*0.01
Group V	vs.	Group II	7.54	4.03	11.04	*0.00
Group IV	vs.	Group III	-1.72	-5.29	1.84	0.66
Group V	vs.	Group III	1.23	-2.27	4.74	0.86
Group V	vs.	Group IV	2.95	-0.55	6.46	0.14

* Significant differences between groups

Figure 14.

Bar Plots with 95% Confidence Intervals of MPa Score by Group

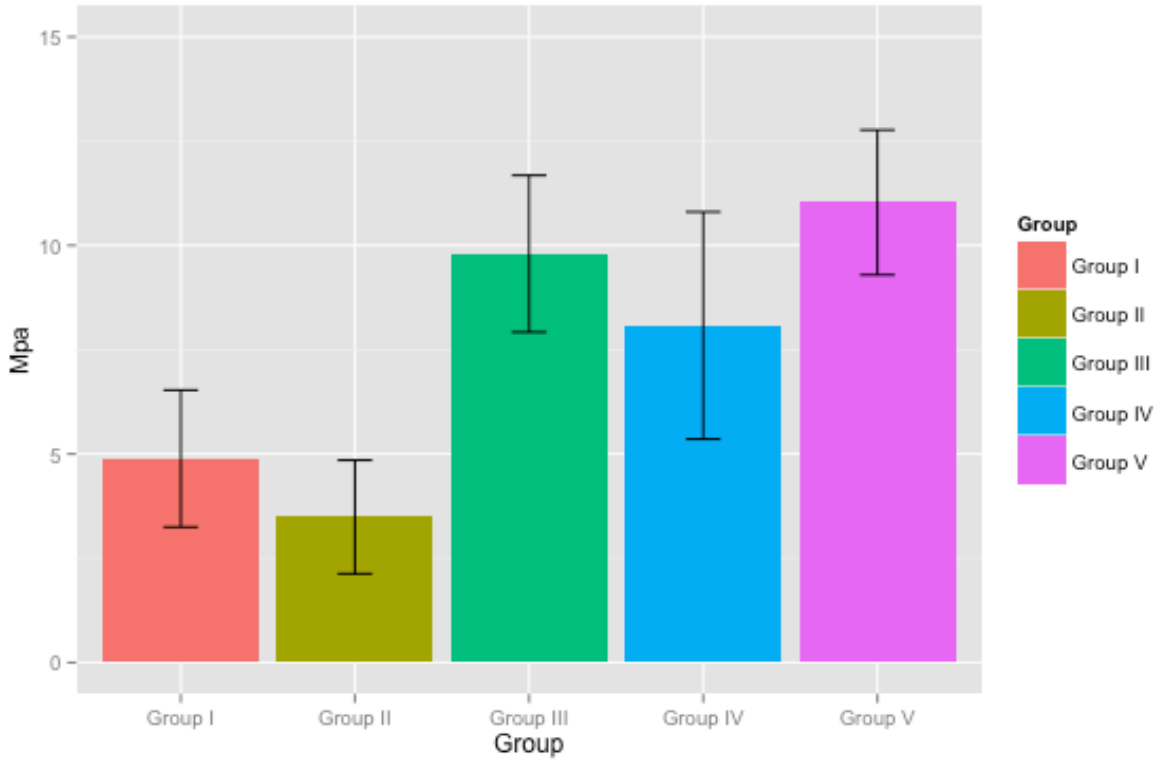


Figure 15.

Bar Plot of ARI Scores by Group

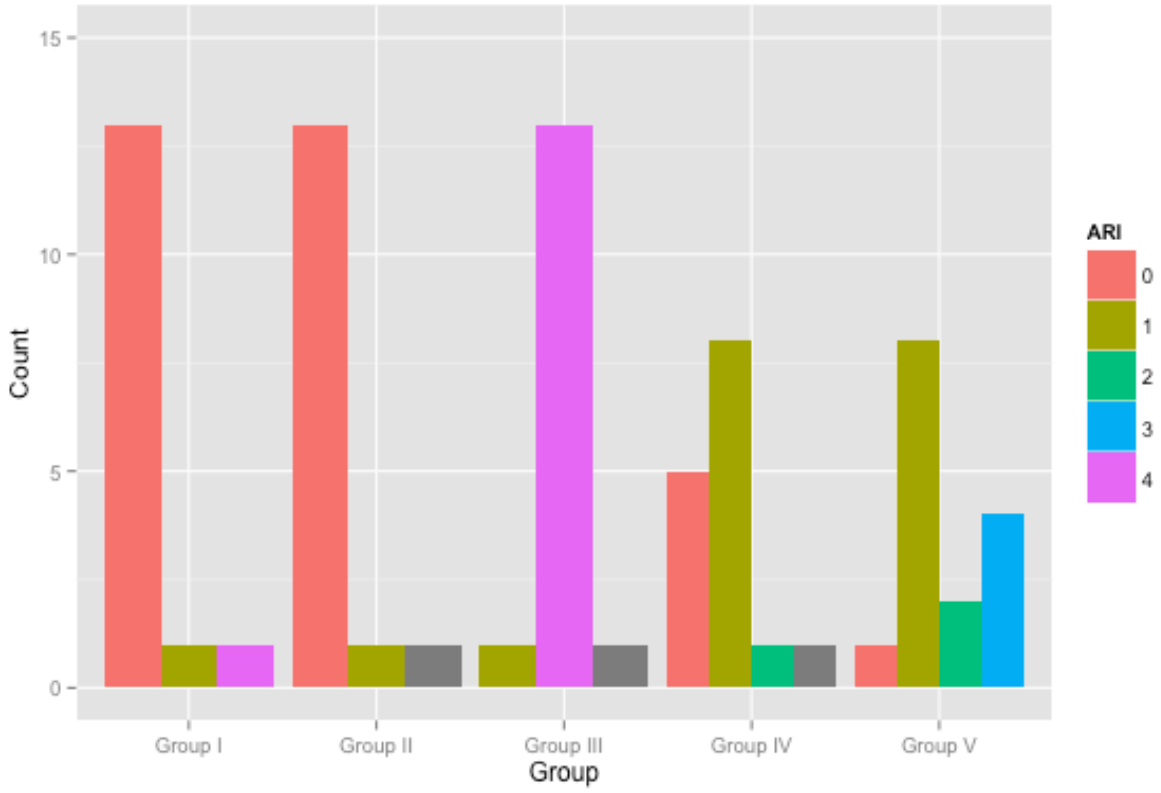
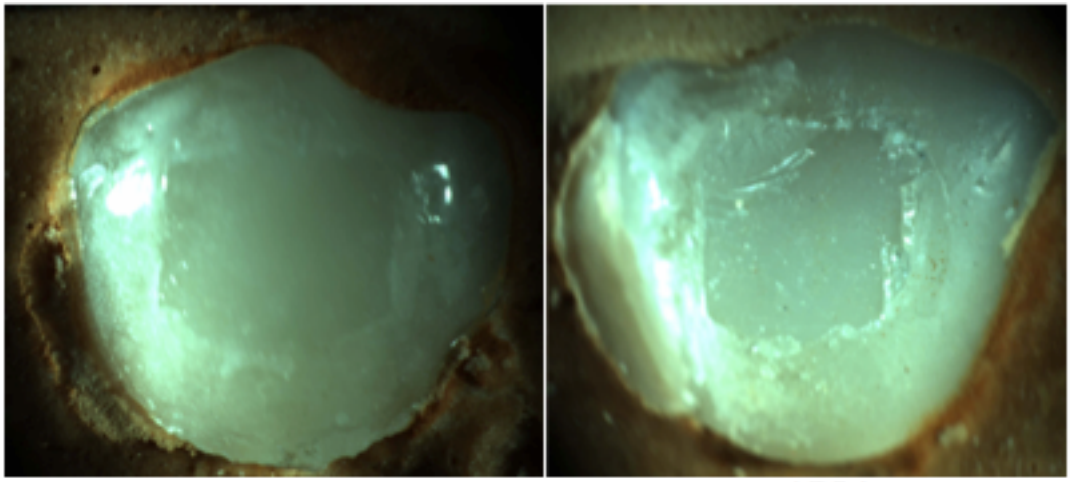


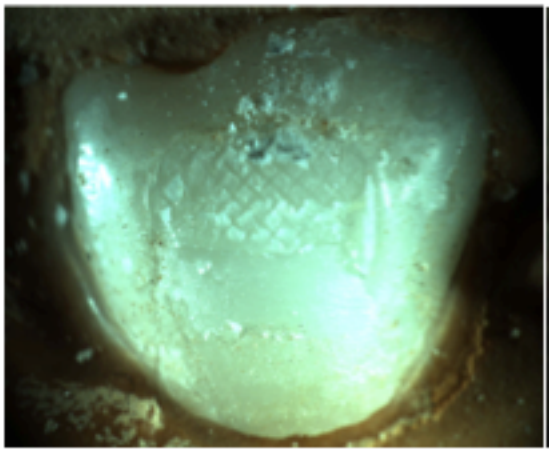
Figure 16.

ARI Assessment



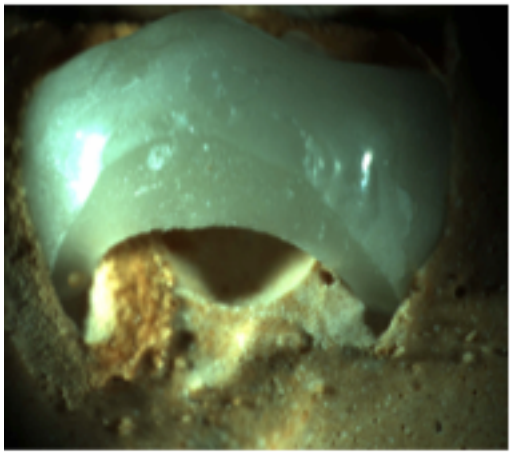
ARI 0

ARI 1



ARI 2

ARI 3



ARI 4

Chapter 4: Discussion

The development of adhesive bonding in orthodontics offers the advantages of efficient chair time, superior esthetics, and improved oral hygiene conditions. Bonding orthodontic attachments has thus become preferred over banding teeth. The increase in adult patients seeking orthodontic care has lead to situations where practitioners may be required to bond attachments to restorative surfaces such as porcelain and gold. While classical restorations have better defined bonding protocols, the bonding protocols for newer restorative materials are unclear. The aim of this study was to determine an effective protocol for bonding attachments to zirconia, a metal-oxide restorative material that has recently gained popularity.

The results showed that the type of restoration and surface preparation had a significant effect on shear bond strength. The highest mean shear bond strength (11.03 MPa) was observed in the group of zirconia crowns microetched with Al_2O_3 particles. These results are consistent with those reported by other studies[25-27] that showed microetch followed by application of an MDP containing primer or resin, produced significantly higher shear bond strength than other surface preparations when bonding to zirconia. Microetch provides a micro-retentive surface by roughening the zirconia, and then application of an MDP adhesive produces a chemical reaction, which results in a bond between the zirconia substrate and adhesive. In accordance with other studies[27, 42], this study demonstrated that silane coupler application was unnecessary to achieve acceptable shear bond strength to zirconia. The microetch/zirconia group also showed significantly more scores of ARI 1, 2, and 3, with 53% of the samples demonstrating an ARI 1 score, indicative of an adhesive type of bond failure. Whitlock[40] reported optimal

shear bond strength to feldspathic porcelain to be 6-8 MPa. Considering the mean shear bond strength and ARI, this study showed that optimal shear bond strength to zirconia might be higher than feldspathic porcelain.

A comparison of mean shear bond strength across groups showed no significant difference between zirconia that was microetched with Al_2O_3 or chemically etched with hydrofluoric acid followed by silane coupler application. These findings are contrary to a study by Derand and Derand[43] who found that hydrofluoric acid had no improvement on the retention of resin cement when bonding to zirconia. They suggested that although hydrofluoric acid did not improve shear bond strength in their study, hydrofluoric acid might alter the adhesive capacity or change the potential free energy of the zirconia surface. Another study by Blatz *et al*.[44] proposed that silanes do not chemically alter the surface of high alumina ceramics, rather they facilitate bonding by increasing the surface wettability in preparation for the resin composite. The wettability property of silanes may influence the adhesive bonding to zirconia as well, in which no chemical reaction occurs between silane and zirconia. This may explain the current findings that suggest hydrofluoric acid followed by silane produced acceptable shear bond strength to zirconia. The zirconia/hydrofluoric acid group was the only group with a mean shear bond strength (8.08 MPa) that fell within the recommended range of 6-8 MPa. This group also showed a favorable adhesive failure debond pattern with 57% of the samples demonstrating an ARI score of 1.

The current study showed sufficient mean shear bond strength (9.80 MPa), when leucite-reinforced porcelain was etched with hydrofluoric acid. This is in contrast to a study by Karan *et al*.[22] that reported hydrofluoric acid to have a significantly lower shear

bond strength to leucite-reinforced porcelain. However, the ARI score for this surface preparation resulted in irreparable fracture for 93% of the samples, an unacceptable result. This result is consistent with a study by Bourke *at al*.[16] that showed hydrofluoric acid to provide adequate shear bond strength yet caused significantly more damage at debond than other surface preparations. Gillis *et al*.[20] reported that higher shear bond strengths were noted with hydrofluoric acid preparation even though the microscopic evaluation of the etched porcelain showed minimal changes to the surface. Based on these results, the current study does not recommend the use of hydrofluoric acid etch when bonding to leucite-reinforced porcelain.

The two groups that demonstrated the lowest mean shear bond strength (3.40 MPa, 4.89 MPa) were the zirconia and leucite-reinforced porcelain prepared with phosphoric acid. In contrast to this study's findings, the mean shear bond strength was considerably lower than reported in other studies[14, 16, 18], which found phosphoric acid to provide adequate bond strength to feldspathic porcelain with the use of a silane primer. The current study showed the shear bond strength of phosphoric acid was significantly different than hydrofluoric acid, which was in contrast to reports by Larmour *et al*.[21]. These disparate results could be explained by the differences in composition of feldpapthic and leucite-reinforced porcelain. Studies showed that phosphoric acid removes the surface glaze, reduces the surface alkalinity, and increases the feldspathic surface free energy in preparation for the resin or adhesive[16]. These effects might not be as potent when preparing leucite-reinforced porcelain, which contains less silica than feldspathic porcelain. The size, number, and distribution of leucite crystals within different ceramics have an effect on the etching patterns of acids[45].

Studies have shown that *in-vitro* findings should not be directly correlated with *in-vivo* conditions. Adequate simulation of the oral cavity can be difficult in laboratory studies, and the composition of different ceramics can vary[46]. Andreasen and Steig[3] found a 48%-52% difference in shear bond strength between *in-vitro* and *in-vivo* conditions. Therefore, it is important to distinguish and take caution when interpreting the results of this *in-vitro* study.

Chapter 5: Conclusion

Shear bond strength was significantly different for type of restorative material and type of surface preparation. Microetch with 50μ Al_2O_3 particles in combination with an MDP containing universal adhesive primer provided the optimal mean shear bond strength, along with favorable debond patterns when bonding to zirconia. Hydrofluoric acid etch in combination with a silane primer provided acceptable shear bond strength to zirconia and was not significantly different from zirconia prepared with microetch; either method can be employed. Leucite-reinforced porcelain prepared with hydrofluoric acid and silane primer resulted in a fracture of 93% of the samples. The use of hydrofluoric acid is therefore not recommended when bonding to leucite-reinforced porcelain.

Appendix: Experimental Data

Group I

Sample	Force (N)	Force (MPa)	ARI
1.1	87.46	7.84	0
1.2	130.75	11.72	0
1.3	15.08	1.35	0
1.4	25.59	2.29	0
1.5	68.97	6.18	4
1.6	108.97	9.76	0
1.7	48.68	4.36	0
1.8	51.58	4.62	0
1.9	49.89	4.47	0
1.10	54.66	4.90	0
1.11	26.73	2.40	0
1.12	32.81	2.94	0
1.13	59.14	5.30	0
1.14	39.14	3.51	1
1.15	19.13	1.71	0

Group II

Sample	Force (N)	Force (MPa)	ARI
2.1	68.42	6.13	0
2.2	32.89	2.95	0
2.3	90.78	8.13	0
2.4	47.27	4.24	0
2.5	18.64	1.67	0
2.6	8.09	0.72	0
2.7	37.99	3.40	0
2.8	48.91	4.38	1
2.9	18.79	1.68	0
2.10	70.53	6.32	0
2.11			
2.12	61.02	5.47	0
2.13	16.85	1.51	0
2.14	11.91	1.07	0
2.15	13.21	1.18	0

Group III

Sample	Force (N)	Force (MPa)	ARI
3.1	81.39	7.29	4
3.2	132.81	11.90	4
3.3	114.79	10.29	4
3.4	98.6	8.84	4
3.5	68.28	6.12	4
3.6	130.48	11.69	4
3.7	118.42	10.61	4
3.8	128.15	11.48	4
3.9			
3.10	126.55	11.34	4
3.11	56.4	5.05	4
3.12	106.58	9.55	4
3.13	202.23	18.12	4
3.14	89.09	7.98	4
3.15	76.9	6.89	1

Group IV

Sample	Force (N)	Force (MPa)	ARI
4.1			
4.2	163.76	14.67	1
4.3	67.78	6.07	0
4.4	139.05	12.46	1
4.5	105.63	9.47	1
4.6	40.24	3.61	1
4.7	33.63	3.01	0
4.8	66.07	5.92	1
4.9	12.19	1.09	0
4.10	144.43	12.94	1
4.11	85.4	7.65	0
4.12	29.11	2.61	0
4.13	180.09	16.14	2
4.14	108.22	9.70	1
4.15	86.13	7.72	1

Group V

Sample	Force (N)	Force (MPa)	ARI
5.1	165.45	14.83	3
5.2	131.63	11.79	1
5.3	110.41	9.89	0
5.4	130.6	11.70	1
5.5	110.29	9.88	1
5.6	152.71	13.68	3
5.7	63.05	5.65	1
5.8	75.94	6.80	1
5.9	174.5	15.64	3
5.10	100.18	8.98	1
5.11	79.70	7.14	1
5.12	100.19	8.98	1
5.13	162.05	14.52	3
5.14	141.04	12.64	2
5.15	148.41	13.30	2

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